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IS 354-2 (1986): Methods of sampling and test for resins for paints, Part 2: Special test methods for alkyd resins [CHD 20: Paints, Varnishes and Related Products]

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IS : 354 (PART 2) - 1986  
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***Indian Standard***  
**METHODS OF**  
**SAMPLING AND TEST FOR**  
**RESINS FOR PAINTS**

**PART 2 SPECIALTEST METHODS FOR ALKYD RESINS**

**( *Second Revision* )**

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Second Reprint OCTOBER 2000

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

**Gr 5**

*February 1987*

**AMENDMENT NO. 4 JUNE 2000  
TO  
IS 354 ( PART 2 ) : 1986 METHODS OF SAMPLING AND  
TEST FOR RESINS FOR PAINTS**

**PART 2 SPECIAL TEST METHODS FOR ALKYD RESINS**

( *Second Revision* )

( *Page 13, clause 11.3, line 18* ) — Substitute '(*see Notes*)' for '(*see Note*)'.

( *Page 13, clause 11.3, Note* ) — Substitute the following for the existing:

**'NOTES**

**1** The precipitate is alcoholate and alcohol of crystallization may be driven off on prolonged heating. However, it is safe to dry up to 60°C for one hour.

**2** For pigmented composition, separation of pigment and varnish may be done as per IS 101 (Part 8/Sec 2)‡ and volatile matter may be determined as per IS 101 (Part 2/Sec 2)§ before proceeding further for determination of phthalic anhydride.'

‡Methods of sampling and test for paints, varnishes and related products : Part 8 Tests for pigments and other solids, Section 2 Pigments and non-volatile matter ( *third revision* ).

§Methods of sampling and test for paints, varnishes and related products: Part 2 Test on liquid paints (chemical examination): Section 1 Water content ( *third revision* ).

**AMENDMENT NO. 3 OCTOBER 1992  
TO  
IS 354 ( Part 2 ) : 1986 METHODS OF SAMPLING AND  
TEST FOR RESINS FOR PAINTS**

**PART 2 SPECIAL TEST METHODS FOR ALKYD RESINS**

*( Second Revision )*

( *Page 13, clause 11.3, line 10* ) — Insert the words 'followed by an ice bath' after the words 'running water'.

( CHD 021 )

**AMENDMENT NO. 2 NOVEMBER 1991  
TO  
IS 354 ( Part 2 ) : 1986 METHODS OF SAMPLING AND  
TEST FOR RESINS FOR PAINTS  
PART 2 SPECIAL TEST METHOD FOR ALKYD RESINS  
( *Second Revision* )**

( *Page 4, clause 4.1, line 2* ) — Substitute 'phenol' for 'phenolphthalein'.

( CHD 021 )

AMENDMENT NO. 1 SEPTEMBER 1989  
TO  
**IS : 354 (Part 2) -1986 METHODS OF SAMPLING AND  
TEST FOR RESINS FOR PAINTS**  
**PART 2 SPECIAL TEST METHODS FOR ALKYD RESINS**  
*(Second Revision)*

(Page 3, clause 0.2, line 7) — Delete the words 'chlorinated rubber'.

(Page 3, clause 0.2, last line) — Delete 'Part 8'.

(CDC 50)

# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

### PART 2 SPECIAL TEST METHODS FOR ALKYD RESINS

#### ( *Second Revision* )

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# *Indian Standard*

## **METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS**

### **PART 2 SPECIAL TEST METHODS FOR ALKYD RESINS**

### *(Second Revision)*

#### **0. F O R E W O R D**

**0.1** This Indian Standard ( Part 2 ) ( Second Revision ) was adopted by the Indian Standards Institution on 18 August 1986, after the draft finalized by the Raw Materials for Paints Industry Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was originally published in 1952 covering methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for method of sampling and test for natural and synthetic resins was published as Part 2 of the above standard in 1971. These two parts were amalgamated and revised in 1976. This revision has been necessitated as more and more newer synthetic resins like polyamides, polyvinyls, chlorinated rubber and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee felt it appropriate to publish this standard in various parts, as indicated below:

- Part 1 General test methods
- Part 2 Special test methods for alkyd resins
- Part 3 Special test methods for phenolic resins
- Part 4 Special test methods for epoxy resins
- Part 5 Special test methods for polyamide resins
- Part 6 Special test methods for amine resins
- Part 7 Special test methods for determination of monomer content in acrylic or vinylacetate containing polymers and emulsions
- Part 8 Special test methods for chlorinated rubber

**0.3** In this standard ( Part 2 ), the test methods covered in 17.1 to 17.7 of IS : 354-1976\* have been included. In addition to above, methods of test for identification of resin, fineness on Hegmann gauge and tolerance to solvent have been added. Methods of tests for chemically modified alkyds will also form a part of this standard as and when made.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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## 1. SCOPE

**1.1** This standard ( Part 2 ) prescribes the methods of test for alkyd resins used in paints and enamels.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard ( Part 2 ), the definitions given in IS : 1303-1963‡ and IS : 6667-1972§ shall apply.

## 3. SAMPLING

**3.1** Representative samples of the alkyd resins shall be drawn as prescribed in 3 of Part 1 of this standard.

## 4. IDENTIFICATION

**4.0** The resin shall be identified by the qualitative tests given below.

**4.1 Phenolphthalein Test** — Heat 1 g of resin with 2 to 3 g of phenolphthalein and 10 drops of concentrated sulphuric acid ( see IS : 266-1977|| ) in a test tube until an orange or brownish-orange melt forms ( see Note ). After the melt has cooled, extract with water, dilute to 1 000 ml and make alkaline with sodium hydroxide solution. Formation of pink colour confirms phthalate and alkyd resin.

NOTE — Too high a temperature or too much acid or too long heating may char the melt.

**4.2 Resorcinol Test** — Heat about 0.5 g of resin with 2 to 3 times the quantity of resorcinol for several minutes. Cool and extract with boiling water. Dilute to 50 to 100 ml with water. Make alkaline with dilute sodium hydroxide solution. A green fluorescence indicates phthalate and alkyd resin.

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\*Methods of sampling and test for resins for paints ( *first revision* ).

†Rules for rounding off numerical values ( *revised* ).

‡Glossary of terms relating to paints ( *second revision* ).

§Glossary of terms used in synthetic resin industry.

||Specification for sulphuric acid ( *second revision* ).

## 5. IDENTIFICATION OF ROSIN IN ALKYD RESINS

**5.0 Outline of the Method** — Presence of rosin is qualitatively determined by any of the following methods.

**5.1 Liebermann-Starch Test** — Dissolve sample by warming/boiling in acetic anhydride. Cool to room temperature. Take one or two ml of this solution in white porcelain dish. Add a drop of sulphuric acid ( sp gr 1.43 ). The presence of violet colour indicates the presence of rosin in the sample.

**5.2 Nichel Modification of Liebermann-Starch Test** — Dissolve 5 ml of sample ( 0.1 to 0.5 g ) in 3 ml of chloroform. Add 5 ml sulphuric acid ( sp gr 1.56-1.58 ) and shake thoroughly. The yellow chloroform will float. After it has become clear, add acetic anhydride drop by drop, the chloroform layer will be coloured violet in presence of trace of rosin. By repeated vigorous shaking, the sulphuric acid layer dissolves the colouring matter and takes on a crimson to purple red colour.

## 6. DETERMINATION OF FINENESS ON HEGMANN GAUGE

**6.0 Outline of the Method** — This test prescribes a method of estimating the fineness of the solution.

### 6.1 Apparatus

#### 6.1.1 *Hegmann Gauge*

**6.2 Procedure** — Place a few drops in the groove of the Hegmann gauge and draw the same along the decreasing thickness direction. The point along the groove at which continuous bitty appearance starts is read in terms of micron scale on the side.

## 7. DETERMINATION OF TOLERANCE TO SOLVENT

**7.0 General** — This test prescribes the extent to which alkyd resins can be diluted. Usually, it is carried out either on volume/volume basis or mass/volume basis or mass/mats basis.

### 7.1 Apparatus

#### 7.1.1 *Graduated Test Tube*

#### 7.1.2 *Beaker*

#### 7.1.3 *Glass Rod*

### 7.2 Procedure

**7.2.1 Volume/Volume Basis** — 1.2 ml of solution of resin is transferred directly into a graduated test tube without touching the sides, and the solvent, for tolerance test, is gradually added in small shots with

immediate shaking to mix the contents of the test tube. The addition is continued till a slight haze is obtained in the solution. The volume of solvent added is noted.

$$\text{Percent tolerance} = \frac{\text{Volume of solvent}}{\text{Volume of resin sample}} \times 100$$

**7.2.2 Mass/Volume Basis** — Method is similar to the above but a weighed quantity of the resin in a graduated cylinder is diluted as above with the solvent, for tolerance. Stop addition when the solution turns hazy. The volume of liquid added is read out from the graduations.

**7.2.3 Mass/Mass Basis** — In a weighed beaker of suitable capacity with a glass rod, weigh accurately 10 g of the alkyd sample. Place the beaker on a white surface with a black dot so that the dot can be seen through the alkyd solution. Add the solvent for thinning and stir after every addition with the glass rod. Add solvent till the dot disappears due to haze in solution. Reweigh the beaker with solution. Find out the weight of solvent added.

$$\text{Percent tolerance} = \frac{\text{Mass of solvent added}}{\text{Mass of resin taken}} \times 100$$

## 8. DETERMINATION OF FATTY ACIDS

**8.0 Outline of the Method** — The resin is refluxed with alcoholic potassium hydroxide and filtered. The filtrate collected and the unsaponifiable and volatile matter are extracted with ether. The aqueous extract is acidified to pH 2, cooled to room temperature and fatty acids present are extracted with ether. The ether extract is evaporated, dried and weighed. The determination is not applicable for alkyd resins containing modifying agents like urea, phenol, melamine, rosin and styrene.

### 8.1 Apparatus

**8.1.1 Beakers** — of 150- and 400-ml capacities.

**8.1.2 Filter Flask**

**8.1.3 Funnel** — long stem, 75 mm diameter.

**8.1.4 Flask and Condenser** — 500-ml, long-necked, fitted with a reflux condenser. The joints between the flask and the condenser shall be ground-glass joints.

**8.1.5 Guard Tube** — of soda-lime.

**8.1.6 Nitrogen Gas Supply**

**8.1.7 Separating Funnels** — three, of 500-ml capacity each.

**8.1.8 Drying Oven** — Vacuum type, controlled thermostatically to maintain a temperature of  $60 \pm 2^{\circ}\text{C}$ .

## 8.2 Reagents

**8.2.1 Alcohol-Benzene Mixture** — Mix 1 volume of absolute alcohol ( *see IS : 321-1964\** ) with 3 volumes of benzene ( *see IS : 1840-1961†* ).

**8.2.2 Benzene** — conforming to IS : 1840-1961†.

**8.2.3 Ether**

**8.2.4 Hydrochloric Acid** — relative density 1.16, conforming to IS : 265-1976‡.

**8.2.5 pH Indicator Papers** — universal type.

**8.2.6 Alcoholic Potassium Hydroxide Solution**

**8.2.7 Sodium Sulphate** — anhydrous, conforming to IS : 255-1982§.

## 8.3 Procedure

**8.3.1** Weigh sufficient resin or resin solution to yield 0.8 to 1.2 g of potassium alcohol phthalate into a long-necked flask. Add 50 ml of benzene. Warm, if necessary, over a hot water-bath to dissolve. Add 60 ml of alcoholic potassium hydroxide and fit the reflux condenser. Place the flask in water-bath, immersing to the level of contents of the flask. Gradually, raise the temperature and reflux for  $1\frac{1}{2}$  h. At the end of this period, remove the flask from bath and wash the inside of the condenser with a few millilitres of alcohol-benzene mixture. Remove the condenser, close the flask with soda-lime guard tube and cool in running water. Cool and filter immediately and quickly through previously tared G 4 sintered glass crucible. Wash the precipitate with benzene-alcohol mixture until the washings are not alkaline to phenolphthalein. Avoid suction of air through the precipitate as the crystals are hygroscopic. Pour about 25 ml of ether and filler. Transfer the filtrate and washings to a 500-ml beaker using water. Concentrate on steam bath to a volume of nearly 25 ml under a blanket of nitrogen gas to avoid oxidation of fatty acids. Transfer the solution to a 500-ml separating funnel with the aid of water and add 300 ml of water and 10 ml of alcohol. Extract the unsaponifiable matter and volatile thinners with successive 50-ml portions of ether, combining the ether extracts in the first separating funnel and using the other two for successive extractions ( *see Note* ). Wash the ether extracts with three 10-ml portions of water,

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\*Specification for absolute alcohol ( *revised* ).

†Specification for benzene, reagent grade.

‡Specification for hydrochloric acid ( *second revision* ).

§Specification for sodium sulphate, anhydrous ( technical grade ) ( *second revision* )

adding water to the main aqueous phase. Discard the combined ether extracts.

NOTE — If the layers do not separate distinctly, draw the aqueous layer carefully and add 2 to 3 ml of alcohol to the emulsion phase. Swirl gently to break emulsion and draw the aqueous layer. This procedure may be repeated on subsequent extractions, if necessary.

**8.3.2** Acidify the aqueous phase with hydrochloric acid, cooling while addition is carried out and bring the solution to pH 2. Cool the mixture to room temperature and extract the fatty acids with successive 25 ml portions of ether until a colourless extract is obtained, combining the ether extracts in the first funnel and using the others for subsequent extractions. Wash the combined ether extract with water until it is free from mineral acids as revealed by indicator paper. Preserve the aqueous phase for polyol determination ( see 9 and 10 ). Dry the combined ether extract in the separating funnel by the addition of anhydrous sodium sulphate. Filter the ether extract through rapid low ash filter paper and collect the filtrate into a previously weighed 150-ml beaker containing a few pieces of porcelain bits and previously weighed to the nearest 1 mg. The ether extract should be decanted from the top opening of the separating funnel. Evaporate the ether portion-wise by placing the beaker and its contents on the water-bath. Cover the filter funnel with a watch-glass during evaporation and maintain an inert atmosphere over beaker by passing nitrogen gas. Remove last portions of fatty acids from sodium sulphate by washing with small amounts of ether until a colourless extract is obtained. Remove the final traces of fatty acids from filter paper with small portions of ether.

**8.3.3** Complete the evaporation of fatty acid-ether solution on the steam-bath while maintaining nitrogen atmosphere. Remove final traces of ether by heating for successive 20 minutes period in vacuum oven at 60°C until constant mass is obtained.

**8.4 Calculation** — Calculate total fatty acids content as follows:

$$\text{Total fatty acids, percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

$M_2$  = mass in g of the beaker plus residue,

$M_1$  = mass in g of the beaker, and

$M$  = mass in g of the material taken for test.

#### 9. DETERMINATION OF GLYCEROL AND ETHYLENE GLYCOL

**9.0 Outline of the Method** — The method covers the determination of glycerol and ethylene glycol in alkyd resins and resin solutions. Other polyhydric alcohols that can be oxidized by periodic acid to formaldehyde

or formic acid or both, will interfere with the determination of glycerol and ethylene glycol. Urea, melamine and phenolic resins interfere. Glycerol and ethylene glycol are determined by adding periodic acid to the aqueous extract obtained in the determination of fatty acids ( 8.3 ). This is titrated against standard sodium hydroxide and further with standard thiosulphate after titrating the iodine liberated by addition of potassium iodide. The primary hydroxyl groups of ethylene glycol and glycerol are oxidized to formaldehyde by periodic acid; the secondary hydroxyl group of glycerol is oxidized to formic acid. By acidimetric and iodometric titration, the proportions of formic acid and formaldehyde may be determined and calculated to glycerol and ethylene glycol by algebraic equations.

## 9.1 Apparatus

**9.1.1 Burettes** — of 50- and 100-ml capacities, conforming to IS : 1997-1982\*.

**9.1.2 Pipettes** — of 20- and 50-ml capacities ( see IS : 1117-1975† ).

**9.1.3 Beakers** — two, each of 400-ml capacity ( see IS : 2619-1971‡ ).

**9.1.4 Long-Necked Flask** — four, each of 1 000-ml capacity.

**9.1.5 Volumetric Flasks** — two, each of 100-ml capacity ( see IS : 915-1975§ ).

**9.1.6 Graduated Cylinder** — 10-ml capacity.

## 9.2 Reagents

**9.2.1 Methyl Purple Indicator** — 0.1 percent.

**9.2.2 Periodic Acid** — Dissolve 11 g of periodic acid crystals in water and dilute to 1 000 ml. Store the solution in brown bottles.

**9.2.3 Potassium Iodide Solution** — Dissolve 200 g of potassium iodide in water and dilute to 1 000 ml.

**9.2.4 Standard Sodium Hydroxide Solution** 0.1 N.

**9.2.5 Standard Sodium Thiosulphate Solution** — 0.2 N.

**9.2.6 Starch Solution**

**9.2.7 Sulphuric Acid** — 1 : 5 ( v/v ). Mix one volume of concentrated sulphuric acid ( conforming to IS : 266-1977|| ) with 5 volumes of water.

\*Specification for burettes ( second revision ).

†Specification for one-mark pipettes ( first revision ).

‡Specification for glass beakers ( first revision ).

§Specification for one-mark volumetric flasks ( first revision ).

||Specification for sulphuric acid ( second revision ).

**9.3 Procedure** — Transfer the aqueous phase obtained from the fatty acid determination ( 8.3 ) to a 400-ml beaker and evaporate to reduce to a volume of 60 ml. During evaporation, keep the mouth of the beaker covered with a watch-glass. Cool to room temperature and filter through a rapid filter paper into a 100-ml volumetric flask, make up to mark and homogenize by shaking. Pipette 20 ml into a 1 000-ml long-necked flask. Add a few drops of indicator and neutralize with sodium hydroxide solution. Add exactly 50 ml of periodic acid into the flask, stopper and swirl well to mix the contents thoroughly. Simultaneously, prepare two blanks containing 20 ml of water and allow to attain the room temperature. Add to the aliquots of the sample and the blank, 100 ml of water and 3 drops of methyl purple indicator and titrate against standard sodium hydroxide. To another aliquot of the filtered solution, add 150 ml of water, 30 ml of potassium iodide solution and 25 ml of sulphuric acid. Titrate the solution against standard sodium thiosulphate solution to faint iodine colour. Add about 5 ml of starch solution and finish the titration with the disappearance of blue colour ( *see Note 2* ). Carry out a blank under similar conditions with all reagents except the sample.

NOTE 1 — The aliquot should be so chosen, if possible, that 15 to 20 percent of periodic acid is consumed during oxidation, considerable periodic acid is required to complete the oxidation and in case more than 20 percent is consumed, the results should be disregarded and a smaller aliquot taken. On the other hand, too small an aliquot is not advisable, for in such a situation, the difference between titration and blank is small.

NOTE 2 — If the end-point is not stable, add water and titrate to a stable end point.

**9.4 Calculation** — Calculate the percentage of glycerol and ethylene glycol as follows:

a) Glycerol and ethylene glycol 
$$(A) \text{ percent by mass} = \frac{(V_1 - V) N \times 0.023015}{M \times F} \times 100$$

where

$V_1$  = volume in ml of standard sodium thiosulphate solution required for blank,

$V$  = volume in ml of standard sodium thiosulphate solution required for sample,

$N$  = normality of standard sodium thiosulphate solution,

$M$  = mass in g of the material taken for test, and

$F$  = aliquot in g fraction of the solution used.

$$\text{b) Glycerol, percent by mass (B) } = \frac{(V_1 - V_2) N \times 0.09206}{M \times F} \times 100$$

where

$V_1$  = volume in ml of standard sodium hydroxide solution used for titration of the sample,

$V_2$  = volume in ml of standard sodium hydroxide solution required for blank,

$N$  = normality of the standard sodium hydroxide solution,

$M$  = mass in g of the sample taken for test, and

$F$  = aliquot fraction.

c) Ethylene glycol, percent by mass = 1.348 (  $A - B$  )

NOTE—In practice, samples containing no ethylene glycol have given values of up to one percent and hence samples giving ethylene glycol values of 1 percent or less should be considered as having no ethylene glycol.

## 10. DETERMINATION OF PENTAERYTHRITOL

**10.0 Outline of the Method** — Monopentaerythritol reacts with benzaldehyde to form dibenzal, a crystalline compound, and determination carried out gravimetrically.

### 10.1 Apparatus

**10.1.1 Beakers** — 100-ml capacity.

**10.1.2 Pipettes**

**10.1.3 Desiccator**

**10.1.4 Oven** — capable of maintaining a temperature of 105°C.

### 10.2 Reagents

**10.2.1 Benzaldehyde-Methanol Reagent** — Add 100 ml of methanol ( conforming to IS : 517-1967\* ) to 20 ml of benzaldehyde.

**10.2.2 Hydrochloric Acid** — conforming to IS : 265-1976†.

**10.2.3 Methanol-Water Mixture** — 1 : 1 ( v/v ). Mix equal volumes of methanol and water. Store one-fourth of the solution in refrigerator and the remainder at room temperature.

\*Specification for methanol ( methyl alcohol ) ( *first revision* ).

†Specification for hydrochloric acid ( *second revision* ).

**10.3 Procedure** — Transfer an aliquot of the solution ( 9.3 ) containing 0.15 to 0.55 g of pentaerythritol to a 100-ml beaker. Reduce the volume of the solution over a water-bath to about 5 ml. To the hot solution, add 15 ml of benzaldehyde-methanol reagent and 12 ml of hydrochloric acid. Mix well and let stand at room temperature for about 15 minutes, swirling occasionally to prevent the precipitate from sticking to the bottom of the beaker. Place the beaker in an ice-bath for about an hour and maintain the temperature at 0 to 2°C. Remove the beaker from ice-bath and filter the contents immediately through a weighed fritted glass crucible of medium porosity. Rinse the beaker with 25 ml of cold methanol-water mixture ( 10.2.3 ) and add to crucible. Continue to transfer and wash the precipitate with about 100 ml of methanol-water mixture at 20 to 25°C as follows:

Disconnect suction or vacuum, pour 10 ml of the methanol-water mixture from beaker to crucible and stir the precipitate to get a homogenous slurry, using a short flat-end glass rod. Start filtering using suction or vacuum. Repeat washing thus about six times. Finally wash the precipitate and rinse the walls of the crucible with 30 ml of methanol-water mixture. Filter completely and dry the precipitate at about 105°C for about 2 hours. Cool in a desiccator and weigh.

#### 10.4 Calculation

$$\text{Monopentaerythritol } P, \text{ percent by mass} = \frac{(M_2 + 0.0269) \times 43.6}{M_1 \times F}$$

where

$M_2$  = mass in g of the precipitate,

$M_1$  = mass in g of the material taken for test, and

$F$  = the aliquot fraction taken for determination.

NOTE — The percentage of monopentaerythritol may be converted to percentage of commercial monopentaerythritol on the assumption that the commercial grade contains 85 percent monopentaerythritol, using the following formula:

Commercial monopentaerythritol, percent by mass =  $P/0.85$ , where  $P$  is the value obtained in 10.4.

### 11. DETERMINATION OF PHTHALIC ANHYDRIDE

**11.0 Outline of the Method** — The method covers the determination of phthalic anhydride content in alkyls and alkyd resin solutions including those containing styrene. The material is refluxed with alcoholic potassium hydroxide solution and phthalic anhydride precipitated as potassium alcohol phthalate and determined gravimetrically.

## 11.1 Apparatus

**11.1.1 Flask and Condenser** — A 500-ml long-necked flask fitted with a 75-cm long air condenser. The joint between the flask and the condenser shall be a taper ground-glass joint.

**11.1.2 Desiccator** — with concentrated sulphuric acid as desiccant.

## 11.2 Reagents

**11.2.1 Benzene**

**11.2.2 Alcoholic Potassium Hydroxide Solution** — Dissolve 66 g of potassium hydroxide in 1 000 ml of absolute alcohol ( conforming to IS : 321-1964\* ).

**11.2.3 Ether** — anhydrous, conforming to IS : 336-1973†.

**11.2.4 Alcohol-Benzene Wash Solution** — 1 : 3 ( v/v ).

**11.2.5 Hydrochloric Acid** — 0.1 N.

**11.3 Procedure** — Weigh from a closed container, a sample of the resin or resin solution sufficient to yield 0.8 to 1.2 g of potassium alcohol phthalate into a 500-ml long-necked flask. Add 150 ml of benzene, warm if necessary over a water-bath and bring into solution. Add 60 ml of alcoholic potassium hydroxide solution. Reflux for 1½ hours over a water-bath using the condenser. Remove the flask from the water-bath, rinse down the inside of the condenser with a few millilitres of alcohol-benzene wash solution. Remove the condenser and stopper the flask with soda-lime guard tube and cool the flask by means of running water. Filter the contents through a weighed fritted glass crucible, when cool. Use alcohol-benzene wash solution to transfer the precipitate completely from the flask to crucible. Wash the precipitate with successive portions of alcohol-benzene wash solution until a few millilitres of wash solution shows no signs of alkalinity to phenolphthalein. Do not allow to draw air through the crystals as they are hygroscopic. Finally wash the precipitate with 25 ml of ether. Wipe the outside of the crucible with a clean cloth and place in an oven at 60°C for one hour ( see Note ). Cool to room temperature in a desiccator and weigh.

NOTE — The precipitate is alcoholate and alcohol of crystallization may be driven off on prolonged heating. However, it is safe to dry up to 60°C for one hour.

## 11.4 Calculation

$$\text{Phthalic anhydride, percent by mass} = \frac{M_1 \times 0.5136}{M_2} \times 100$$

where

$M_1$  = mass in g of the precipitate obtained, and

$M_2$  = mass in g of the material taken for test.

\*Specification for absolute alcohol ( revised ).

†Specification for ether ( second revision ).

## 12. DETERMINATION OF PHTHALIC ANHYDRIDE CONTENT IN ALKYDS CONTAINING OTHER DIBASIC ACIDS

**12.0 Outline of the Method** — The method is based on spectrophotometry. It is suitable for the determination of phthalic anhydride content in alkyds containing dibasic acids like maleic, fumaric, adipic acids, etc. The material is saponified in alcoholic potassium hydroxide and benzene and the potassium salt of phthalic acid precipitated. With the exception of maleic and fumaric acids, other interfering substances are eliminated by dissolving the precipitate in water, adjusting the pH to 2.5 with nitric acid and filtering. Phthalic acid is then reprecipitated as non-atoichiometric lead phthalate and calculated to phthalic anhydride, using a factor obtained when compositions of known purity are analyzed.

### 12.1 Apparatus

**12.1.1 Flask and Condenser** — A 500-ml long-necked flask fitted with 76-cm long air condenser, having tapered ground-glass joint.

**12.1.2 Oven**

**12.1.3 Desiccator** — with sulphuric acid as desiccant.

### 12.2 Reagents

**12.2.1 Benzene** — anhydrous.

**12.2.2 Alcoholic Potassium Hydroxide Solution** — same as in **11.2.2**.

**12.2.3 Ether** — anhydrous, conforming to IS : 336-1973\*.

**12.2.4 Alcohol-Benzene Wash Solution** — 1 : 3 (v/v).

**12.2.5 Glacial Acetic Acid** — conforming to IS : 695-1986†.

**12.2.6 Methanol** — anhydrous, conforming to IS : 517-1967‡.

**12.2.7 Lead Acetate Solution** — Dissolve 25 g of lead acetate trihydrate in glacial acetic acid, dilute to 100 ml with acetic acid.

**12.2.8 Nitric Acid** — 1:3 (v/v). Mix one volume of nitric acid conforming to IS : 264-1976§ with 3 volumes of water.

**12.3 Procedure** — Weigh sufficient amount of material from closed container into a 500-ml long-necked flask and follow the digestion and filtration processes as prescribed in **11.3**. Then dissolve the contents of the crucible in 70 ml of water using a filter so as to collect the washings in a 250-ml beaker. Adjust the pH of the contents to 2.5 by

\*Specification for ether (second revision).

†Specification for acetic acid (third revision).

‡Specification for methanol (methyl alcohol) (first revision).

§Specification for nitric acid (second revision).

adding nitric acid. Let stand the contents for 30 minutes and then filter through a double thickness fine filter paper directly into a 100-ml volumetric flask (see Note). Make up to mark with water, using water simultaneously to wash the beaker and filter. Stopper the flask and shake well. Withdraw an aliquot containing not less than 60 mg or not more than 90 mg of the dissolved salts. Transfer this to a 250-ml long-necked flask. Dry the contents of the flask in an oven at 60°C. Add 5 ml of glacial acetic acid, vent the stopper by inserting a paper strip under one side, and heat in the oven at 60°C for one hour. Add 100 ml of anhydrous methanol and continue heating in the oven with occasional agitation until the crystals are completely dissolved. To the hot solution add from a pipette 2.0 ml of lead acetate solution, agitating while adding. Return the flask to oven at 60°C for one hour. Remove the stopper tightly after 30 minutes. Allow to stand for at least 12 hours. Filter through fritted glass crucible of medium porosity. Wash the flask with anhydrous methanol and transfer the washings to the filter. Carefully examine the filtrate. If cloudy, filter again. Dry the crucible for 1 hour at 105°C, cool in a desiccator and weigh.

NOTE — If the sample does not cloud when acidified, it may be diluted to volume at once and the filtering omitted.

## 12.4 Calculation

$$\text{Phthalic anhydride, percent by mass} = \frac{M_1 \times 0.323}{M_2} \times 100$$

where

$M_1$  = mass in g of lead precipitate, and

$M_2$  = mass in g of the material present in the aliquot used.

## 13. DETERMINATION OF UNSAPONIFIABLE MATTER

**13.0 Outline of the Method** — The material is refluxed with sodium hydroxide and unsaponifiable matter separated, evaporated and weighed. The method is not applicable to alkyd resins containing modifying agents like urea, melamine and phenol.

### 13.1 Apparatus

**13.1.1 Beaker** — of aluminium and capacity 125-ml.

**13.1.2 Flask and Condenser** — same as in 12.1.1.

**13.1.3 Separating Funnels** — three, each of 500-ml capacity.

**13.1.4 Drying Oven** — with thermostatic control to operate at  $80 \pm 5^\circ\text{C}$ .

## 13.2 Reagents

**13.2.1 Benzene** — conforming to IS : 1840-1961\*.

**13.2.2 Benzene-Alcohol Mixture** — Mix equal volumes of benzene ( conforming to IS : 1840-1961\* ) and ethyl alcohol ( conforming to IS : 323-1959† ), add 2 drops of phenolphthalein indicator solution and neutralize to a persistent faint pink colour with sodium hydroxide solution.

**13.2.3 Ethyl Alcohol** — 95 percent by volume ( conforming to IS : 323-1959† ).

**13.2.4 Ether** — conforming to IS : 336-1973‡.

**13.2.5 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit ( conforming in IS : 323-1959† ).

**13.2.6 Sodium Hydroxide Solution** — Dissolve sodium hydroxide in equal mass of water.

**13.2.7 Standard Sodium Hydroxide Solution** — 0.02 N.

**13.2.8 pH Papers**

**13.3 Procedure** — Weigh sufficient material so as to contain 0.05 to 0.2 g of unsaponifiable matter into a 200-ml long-necked flask. Add 10 ml of benzene, and warm to dissolve the sample. Add 50 ml of alcohol, swirl to mix, add slowly 5 ml of sodium hydroxide solution and 5 ml of water. Attach the condenser and reflux gently over a water-bath for 2 hours. Remove from water-bath, cool to room temperature, rinse down the condenser and joint with water. Transfer the contents of the flask completely to a separating funnel, using water. Finally, rinse the flask with three 25-ml portions of ether, adding the ether washings to the separating funnel. Add sufficient water to bring the volume to 300 ml and add 10 ml of alcohol. Stopper the separating funnel, shake gently and allow the layers to separate. Draw off the lower aqueous layer to a second separating funnel. Continue the extraction of aqueous layer with successive 20-ml portions of ether until a colourless ether extract is obtained, combining the ether extractions in the first funnel. Wash the combined ether extract contained in the first separating funnel with 25 ml of water until the washings are neutral to pH paper. Transfer the ether extract completely into a 125-ml beaker containing a few porous bits, weighed previously. Evaporate the ether over a steam-bath carefully. When all the ether has been removed, transfer the beaker to an oven previously heated to 80°C. Heat to constant mass, cool to room temperature in a desiccator and weigh. After weighing, add 50 ml of

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\*Specification for benzene, reagent grade.

†Specification for rectified spirit ( revised ).

‡Specification for ether ( second revision ).

warm benzene-alcohol mixture. Titrate against standard sodium hydroxide solution to the same persistent faint pink colour as in the neutralization of benzene-alcohol mixture.

### 13.4 Calculation

$$\text{Unsaponifiable matter, percent by mass} = \frac{M_1 - (V \times N \times 0.280)}{M_2} \times 100$$

where

$M_1$  = mass in g of the residue obtained,

$V$  = volume in ml of standard sodium hydroxide used in the titration,

$N$  = normality of standard sodium hydroxide solution, and

$M_2$  = mass in g of the material taken for test.

NOTE — A factor of 0.280 is used in the equation on the assumption that the acid is 18 carbon atom fatty acid. If coconut, lauric, or other short chain acids are suspected, an arbitrary factor 0.216 shall be used, and this fact shall be reported with the test result.

